Advances in Concrete Construction, *Vol. 5, No. 3 (2017) 241-255*
DOI: https://doi.org/10.12989/acc.2017.5.3.241

Evaluation of incorporating metakaolin to evaluate durability and mechanical properties of concrete

Alireza Joshaghani*[1], Mohammad Amin Moeini[2] and Mohammad Balapour[2]

[1]*Zachry Department of Civil Engineering, Texas A&M University, College Station, TX-77840, USA*
[2]*Department of Civil Engineering, Amirkabir University of Technology, Tehran, Iran*

(Received March 14, 2017, Revised May 23, 2017, Accepted May 24, 2017)

Abstract. Concrete is known to be the most used construction material worldwide. The environmental and economic aspects of Ordinary Portland Cement (OPC) containing concrete have led research studies to investigate the possibility of incorporating supplementary cementitious materials (SCMs) in concrete. Metakaolin (MK) is one SCM with high pozzolanic reactivity generated throughout the thermal activation of high purity kaolinite clay at a temperature ranging from 500℃ to 800℃. Although many studies have evaluated the effect of MK on mechanical properties of concrete and have reported positive effects, limited articles are considering the effect of MK on durability properties of concrete. Considering the lifetime assessment of concrete structures, the durability of concrete has become of particular interest recently. In the present work, the influences of MK on mechanical and durability properties of concrete mixtures are evaluated. Various experiments such as slump flow test, compressive strength, water permeability, freeze and thaw cycles, rapid chloride penetration and surface resistivity tests were carried out to determine mechanical and durability properties of concretes. Concretes made with the incorporation of MK revealed better mechanical and durability properties compared to control concretes due to combined pozzolanic reactivity and the filler effect of MK.

Keywords: concrete; metakaolin; mechanical properties; durability; compressive strength; rapid chloride permeability test; surface resistivity

1. Introduction

Portland cement is traditionally known to be the most used binder material in concrete. The incremental trend of concrete usage has led to a large volume of Portland cement production annually around the world. Over 4 billion tons of Portland cement were produced in 2015 around the world from which up to 4 billion tons of CO_2 gas were emitted to the air due to the known fact that production of 1 ton Portland cement would result in 0.9 to 1 ton of CO_2 emissions. In addition, the cement industry is one of the primary consumers of natural resources. The usage of supplementary cementitious materials (SCMs) in concrete has increased over the past decade due to the positive effects of pozzolanic reactions: Consumption of calcium hydroxide or CH (An undesirable result of hydration of cement) and taking part in further C-S-H gel production,

*Corresponding author, E-mail: joshaghani@tamu.edu

http://www.techno-press.org/?journal=acc&subpage=7 ISSN: 2287-5301 (Print), 2287-531X (Online)

enhanced resistance to cracking due to denser microstructure (Joshaghani *et al.* 2016). This is because of remarkably lower heat released through the cement hydration process and better mechanical properties due to lower permeability as a result of further C-S-H gel formation. Fly ash, ground granulated blast furnace slag, rice husk ash, and metakaolin (MK) are some of the most common SCMs incorporated in concrete manufacture yet (Siddique *et al.* 2001).

MK is a cement replacement material, formed when the pure kaolinite is calcined at high temperatures from 500℃ to 800℃. Thermal activation of mineral clay leads to the crystalline structure breakdown (by dehydroxylation) the aforementioned process leads to an aluminosilicate non-crystalline part creation. MK ($Al_2O_3.2SiO_2$) or AS_2, is formed by such a process (Kurt *et al.* 2016, Türkel *et al.* 2009). MK addition to cement mixtures is reported to improve the mechanical properties of concrete. Major influences of MK addition to concrete is reported to be as a result of modifications in hydration products due to the pozzolanic activity of MK. Brykov *et al.* investigated for the effect of highly active MK addition on hydration products of cement (Byrkov *et al.* 2015). The authors have concluded that cement paste produced by MK incorporation contains C-S-H gel with longer aluminosilicate chains. In addition, the ettringite content in cement paste including high levels of MK is higher than that in control samples fabricated only by OPC. The authors have reported that it is a result of the liberation of aluminum ions from MK and their taking part in the reaction to create additional ettringite.

Researchers reported that the compressive strength of mixture increased when using MK (Johari *et al.* 2011, El-Din *et al.* 2017). Rezaifar *et al.* investigated the combined use of MK in concrete. Crumb rubber was incorporated as aggregate replacement material, while MK was used to replace cement. The authors also used Response Surface Method (RSM) in order to optimize the mix designs. Even though the strength of concrete decreased by the incorporation of crumb rubber, MK notably lowered the compressive strength loss by pozzolanic reactions. Furthermore, the results of the water absorption test revealed that use of MK was very effective in decreasing the water absorption. The authors found that the simultaneous usage of crumb rubber by 3.3 vol.% replacement of aggregate and MK by 19.5 vol.% replacement of cement presented the optimized results as the compressive strength was highest and the water absorption was minimized (Rezaifar *et al.* 2016).The dehydroxylation breakdown of kaolinite clay can be obtained by either thermal adjustment or mechanical process of grinding. In a study, Portland cement was replaced by MK at 10% and 20% levels of replacement (by weight), and mortars yielded 5-6% higher strengths when compared to control mixture at 28 days. The authors concluded that a slight increase in strength of mortars containing MK was due to a greater level of agglomeration, which occurred when MK was being activated thermally (Ilić *et al.* 2017). The level of CH in the concrete paste is reported to decrease significantly by the addition of MK. A number of authors have reported that MK addition to concrete can enhance the durability properties of concrete as a result of lower CH content. However, the effectiveness of MK mostly depends on the degree of reactivity of such material which in turns depends on thermal activation temperature, and purity of kaolinite used (Salau *et al.* 2015). Water absorption of mortars containing MK was investigated by Haining *et al.* (Haining *et al.* 2017). The authors have reported that water absorption of the mixture was decreased by the addition of MK, especially at early ages, since the packing density of the mixture was optimized because of the extremely fine MK particles. It was also noted that the water absorption of specimens was reduced by increasing the dosage of incorporation to 10% of ordinary Portland cement (OPC) weight. In addition, the primary products of cement hydration in mixtures were found to be Portlandite and ettringite, while MK decreased the calcium hydroxide content due to the pozzolanic reaction. The durability of MK Self-Compacting Concrete (SCC) was investigated

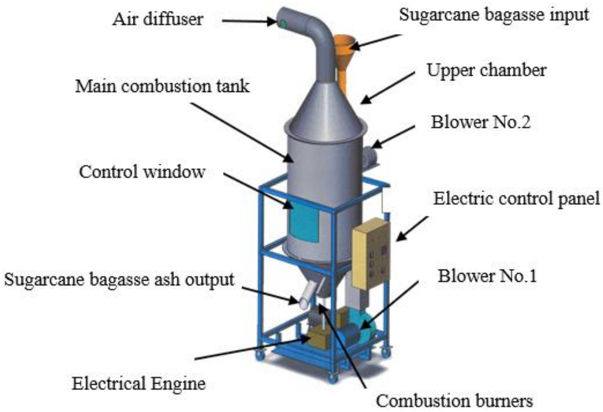


Fig. 1 Schematic image of the furnace used for the burning process (Ramezanianpour *et al.* 2009)

by Badogiannis *et al.* (Badogiannis *et al.* 2015) MK was used to replace cement or limestone powder at various levels. A positive influence on the open porosity was observed for both replacement styles, irrespective of the level of replacement. Also, according to the chloride ion penetration test results, replacement of either cement or limestone powder by MK resulted in a reduction of the non-steady state chloride migration coefficient. A group of researchers found that the use MK significantly decreased the diffusion coefficient of concrete. Also in another study, the authors reported that incorporating up to 15% MK would improve the corrosion resistance and absorption by decreasing penetrability of mixtures (Bu *et al.* 2016, Tafraoui *et al.* 2016).

Today, MK is successfully being used in various types of concrete, such as ultra-high performance concrete, high-performance concrete, self-compacting concrete and fiber reinforced concrete (Siddique *et al.* 2001). The use of MK has yielded technical, environmental and economic benefits to the cement and concrete industry. Considering the abundant kaolin mines in Iran, this study investigates the performance of concrete mixtures containing MK in terms of slump flow test, compressive strength, water penetration, freeze and thaw cycles, rapid chloride permeability test (RCPT) and the surface resistivity at 7, 28, 90 and 180 days. In this study, the MK replacement percentage were 0, 10, 12.5 and 15% by weight of cement at two different water/cementitious-materials (W/CM) ratios.

2. Experimental program

2.1 Material

Type I Portland cement was used in this study, which had chemical and physical properties as shown in Table 1 (ASTM C150/C150M-16e1). To provide high-grade supplementary cementitious materials in terms of pozzolanic activity, the burning conditions such as temperature and the burning time were controlled. A furnace built to burn raw kaolinite clay in the Concrete Technology and Durability Research Center at Amirkabir University is shown in Fig. 1 (Ramezanianpour *et al.* 2009). The burning temperature, the oxygen rate, and the burning time are the relevant parameters in the production of pozzolanic materials. Thus, two fans have been used to supply the required air for burning. The first fan with a power of 3700 W and 2950 cycles per

Table 1 Physical and chemical characteristics of cement and metakaolin

	Physical properties		Chemical analysis (%)							
	Specific Gravity	Blaine, (cm^2/g)	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	Na_2O	K_2O	LOI
Cement	3.16	3080	20.82	4.23	2.61	64.11	3.06	0.20	0.81	2.74
Metakaolin	2.49	3950	69.33	19.42	0.96	4.01	0.41	0.02	0.41	2.41

Table 2 Chemical and mineralogical analyses of kaolin

	Chemical Analysis (%)								Mineralogical analysis (%)			
	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	Na_2O	K_2O	LOI	Kaolinite	Quartz	Calcite	Other
Kaolin	78.12	14.55	0.79	3.15	0.31	0.07	0.75	6.94	61.05	29.11	5.86	3.98

Table 3 Mix proportions of concrete

Group	Mix	W/b	Metakaolin (%)	Metakaolin (Kg/m^3)	Cement (Kg/m^3)	Water (Kg/m^3)	Coarse Aggregate (Kg/m^3)	Fine Aggregate (Kg/m^3)
1	OPC	0.47	0	0	420	197.4	759	948
	MK10	0.47	10	42	378	197.4	759	948
	MK12.5	0.47	12.5	52.5	367.5	197.4	759	948
	MK15	0.47	15	63	357	197.4	759	948
2	OPC	0.37	0	0	420	155.4	802	1007
	MK10	0.37	10	42	378	155.4	802	1007
	MK12.5	0.37	12.5	52.5	367.5	155.4	802	1007
	MK15	0.37	15	63	357	155.4	802	1007

minute was placed in the lower part of the furnace with a maximum flow ventilation of 745 $\frac{m^3}{h}$. The second fan with a power of 75 W and 2900 cycles per minute was installed on the basic framework of a cylindrical body, which brings in the required air from the internal shells to the furnace via the embedded holes on the mixer rods. The maximum output flow of the second fan is 245 $\frac{m^3}{h}$. The temperature inside the furnace is measured by three thermocouples in three different parts of the furnace (Ramezanianpour *et al.* 2009).

To produce MK with suitable pozzolanic activities, different specimens have been burnt in various conditions of time and temperature. According to the previous studies, it was confirmed that burning at the temperature between 500℃ and 800℃ will produce amorphous silica. In addition to temperature change, the duration of ignition was another variable parameter which was studied. Considering the system performance, it was identified that all specimens in different temperatures complete the burning process in 60 minutes.

The chemical composition of MK and mineralogical results of used kaolin are presented in Tables 1 and 2, respectively. Sand with the maximum aggregate size of 4.75 mm and crushed stone with the maximum aggregate size of 19 mm were used (ASTM C33/C33M-16e1). The specific gravity of coarse and fine aggregates was 2610 and 2513 kg/m^3. To get the purposed

Table 4 Specimens geometry

Test Method	Flowability	Compressive Strength	Water Absorption	Capillary Absorption	Freeze and Thaw	Surface Resistivity (SR)	Water Penetration	RCPT
Standard	ASTM C143	ASTM C39	BS 1881-Part 122	RILEM CPC 11.2	ASTM C666	AASHTO TP 95	BS EN-12390-8	ASTM C1202
Specimens Geometry	Slump cone (30×20× 10 cm)	Cube 100 mm	Cube 50 mm	Cube 50 mm	Prism 100×100× 400 mm	Cyllendral 100× 200 mm	150 mm	Cyllendral 100× 50 mm

slump, polycarboxylic acid-based superplasticizer was used.

2.2 Specimens preparation

There are two sets of mixtures, Group 1 and Group 2, which were designed at 0.37 and 0.47 w/cm ratios, respectively, and a constant total cementitious materials of 420 kg/m^3 was used for all mixtures. In this study, the MK replacement percentage were 0, 10, 12.5 and 15% by weight of cement, as shown in Table 3. After casting, the concrete specimens were covered with a plastic sheet under laboratory conditions and they were cured in lime-saturated water after one day at 24±2℃ until the test date.

2.3 Test methods

This section provides the used test method in this study. Table 4 presents each tests' methods specimens condition, including a number of specimens and sample geometry.

2.3.1 Fresh concrete tests

The slump flow test was conducted in conformity with the standard techniques given by ASTM C143 (ASTM C143/C143M-15a). The slump flows were kept constant at 70±10 mm. The superplasticizer was used at slight percentages according to the results obtained for the slumps. The slump flow test was carried out to evaluate the capabilities of MK in the slump retention. The density of the mixtures was obtained by weighing the fresh concrete into a standard mold with a specific volume in accordance with ASTM C 138 standard (ASTM C138/C138M-16a).

2.3.2 Compressive strength

The cube with 100 mm dimension was used for compressive strength. Compressive strength tests were conducted with a loading rate of 0.5 N/mm^2/s at the age of 7, 28, 90, and 180 days. The average compressive strength of three specimens was considered for each age.

2.3.3 Transport tests

There are different methods to evaluate the absorption of concrete samples with MK. One is testing water absorption based on BS 1881-Part 122 (BS 1881-116 1983). The cubic specimens were dried at 45℃ for 14 days to reach the constant weight. Then samples were immersed in water and scaled after 0.5, 1, 24, 72 and 168 hours to measure the weight variation. This method would evaluate water absorption that happened in pores. These tiny voids are emptied by oven drying and occupied again with water after the immersion.

In another approach, capillary absorption is measured through the non-saturated concrete specimens. In this method, a sample is in adjacent with a water layer on one lateral and absorbed water evaporation from the other part, a steady flowing regime through capillary absorption is established (Piasta *et al.* 2017). The test was performed for measuring of capillary water absorption in accordance with RILEM CPC 11.2, TC 14-CPC for testing capillary absorption of MK specimens. The cubic specimens with 100 mm dimensions were dried in the oven at 45±5℃. They were immersed in a water bath with 5 mm depth.

2.3.4 Freeze and thaw

The resistance to freezing/thawing of MK samples was evaluated in accordance with ASTM C 666 (ASTM C666/C666M-15 2015), in which specimens were subjected to repeated freezing and thawing cycles. Specimens were used to measure the fundamental transverse frequency by using the force resonance method. Samples with dimensions of 100×100×400 mm were cured under standard conditions and tested for integrity by recording the relative dynamic modulus of elasticity (Eq. (1)) every 25 cycles up to 300 cycles.

$$P_c = \frac{n_1^2}{n^2} \times 100 \tag{1}$$

P_c: relative dynamic modulus of elasticity, after c cycles of freezing/thawing (%),
n: the fundamental transverse frequency at 0 cycles of freezing/thawing,
n_1: the fundamental transverse frequency after c cycles of freezing/thawing

2.3.5 Surface resistivity (SR)

The surface resistivity was measured by the electrical resistivity of water-saturated concrete for each specimen. Electrical resistivity is a function of moisture and electrolyte content of the pores in concrete, which being measured based on AC impedance spectrometry using a resistance meter (Yousuf *et al.* 2017). Experiments have been done with Wenner 4-probe meter. The probe array spacing used was 40 mm. The resistivity measurements were taken at four quaternary longitudinal locations of the specimen. The likely difference and resulting current can be applied to find the electrical resistance. Three readings were obtained from the data logger for each cylinder specimen. The bulk resistivity was calculated as follows (Eq. (2))

$$\rho = \frac{V}{I} \times \left({}^{A}\!/_{L}\right) = R \times \left({}^{A}\!/_{L}\right) \tag{2}$$

where ρ is the electrical resistivity (KΩ cm), R is bulk electrical resistance (KΩ), A is a cross-sectional area (cm^2), L is the distance between two electrodes (cm), I is measured current, and V is the voltage.

2.3.6 Water penetration

The water penetration test was used to study the permeability of mixture in accordance with BS EN-12390-8. Cubic specimens with 150 mm length were kept under 0.5 Mpa pressure for 72 hours. Then, the specimens were split into two parts. The water penetration profile was obtained with the maximum depth of water penetration in specimens.

2.3.7 Rapid chloride permeability test (RCPT)

The resistance of concrete against the chloride ions penetration was measured by RCPT at the

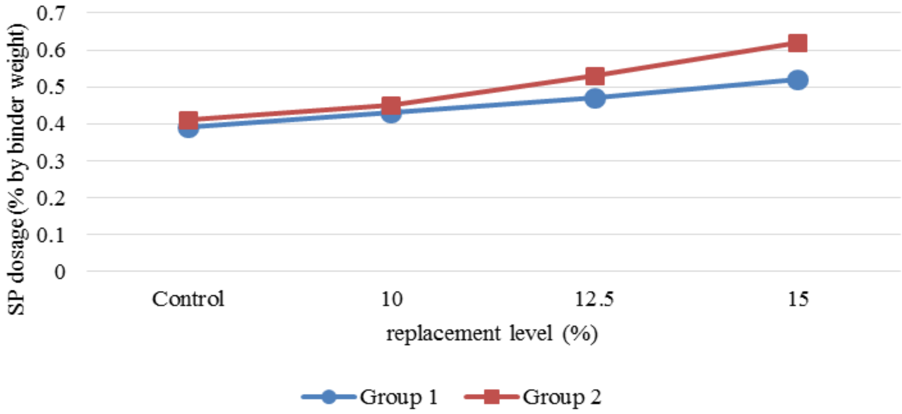


Fig. 2 SP dosage of binary concrete mixes to obtain target fluidity

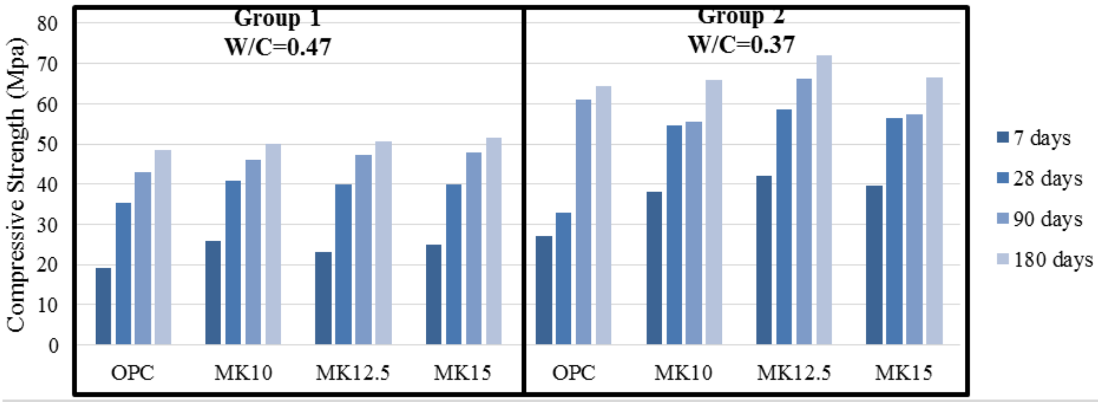


Fig. 3 The effect of metakaolin on the compressive strength at various ages

ages of 7, 28, 90 and 180 days in accordance with ASTM C-1202. Cylindrical specimens with a diameter of 100 mm and thickness of 50 mm were put under a 60-V potential for six hours. The amount of passed charge indicates the chloride ion permeability.

3. Result and discussion

3.1 Superplasticizer (SP) demand

The required SP dosage to achieve the target slump flow of 70±10 mm of all binary mixes is shown in Fig. 2. The superplasticizer was used at low percentages according to the results obtained for the slumps. The SP dosage of the group 2 blends was significantly higher than group 1 and the control concrete. The higher the replacement of cement by MK, the more SP was required to achieve the target slump flow, which probably stemmed from a large amount of pores in the frame structure and high surface area of MK and in turn led to increased water demand. A binder having a greater surface area requires more water to obtain a given slump flow, so more SP is required to keep the content of water constant (Ardalan *et al.* 2017).

Table 5 ANOVA results of compressive strength

Mixtures	Group 1				Group 2			
	OPC	MK10	MK12.5	MK15	OPC	MK10	MK12.5	MK15
Std Dev.	1.85	2.01	1.86	1.23	2.12	1.45	1.31	1.08
Sig.*	-	0.997	0.854	0.035	-	0.624	0.02	0.126

*The mean difference is significant at the 0.05 level

Table 6 Results of water absorption and capillary water absorption versus time

	Mix Design ID	Time (hr)									
		0.5	1	3	24	72	168	24	48	72	168
		Water Absorption (%)						Capillary Water Absorption (mm)			
Group 1	OPC	2.55	3.45	4.38	4.76	5.08	5.57	2.5	4.2	5.4	6.0
	MK10	2.11	3.11	3.86	4.42	4.84	5.15	2.3	3.7	5.0	5.7
	MK12.5	1.84	2.71	3.57	4.02	4.32	4.67	2.2	3.3	4.7	5.3
	MK15	1.73	2.65	3.37	4.01	4.37	4.78	2.0	3.2	4.5	5.2
Group 2	OPC	2.42	3.33	4.27	4.61	4.89	5.37	2.7	4.1	5.3	5.9
	MK10	1.97	2.93	3.75	4.27	4.68	5.04	2.3	3.6	4.9	5.5
	MK12.5	1.65	2.59	3.42	3.90	4.20	4.57	2.2	3.3	4.6	5.1
	MK15	1.54	2.50	3.25	3.82	4.25	4.66	1.9	3.1	4.5	5.0

3.2 Compressive strength

The compressive strengths of concrete specimens with different cement replacement percentage and w/cm are shown in Fig. 3 at the ages of 7, 28, 90, and 180 days. The compressive strength values were developed by the time of curing and decreasing the w/cm ratio. As mentioned in the literature review, the effects of MK in improving the strength are attributed to the dilution effect, filling effects, and the pozzolanic reaction of MK with calcium hydroxide.

In both groups, all the samples with MK had higher compressive strengths, compared to control sample in every age. The strength of the group 2 at the age of 180 days age were higher than that of the control specimen by 14.2%, 21.9%and 14.5% for MK10, MK12.5 and MK15, respectively. The same values for Group 1 were 9.2%, 13.5%, and 18.6%. The reason of compressive strength loss for MK15 compared to MK12.5 was the clinker dilution effect due to the consequence of replacing cement by MK. However, the filler effect in specimens with MK, reacted opposite of the dilution effects. The reason is attributed to the pozzolanic reaction of MK with calcium hydroxide (ASTM C33/C33M-16e1). Based on the results, MK with 15% replacement and 0.47 for w/cm had the greatest compressive strength compared to other samples. The same result was reported by other researchers (Tafraoui *et al.* 2016).

The values of the analysis of variance (ANOVA) are shown in Table 5, which indicate whether the compressive strength difference between samples containing MK and control sample are significant. Based on a defined level of 0.05, when the significance factor of samples containing MK is less than or equal to 0.05, a significant difference exists between its compressive strength with control samples. Otherwise, samples with significance factor greater than 0.05 have an eligible difference with the control sample (Tabatabaeian *et al.* 2017). Therefore, the addition of

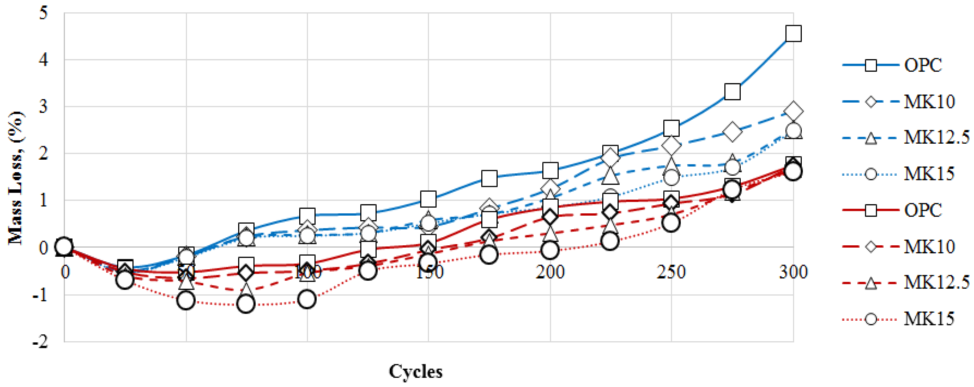


Fig. 4 Mass loss of concrete versus freeze and thaw cycles

Table 7 Dynamic modulus of concrete after the particular freeze and thaw cycles (GPa)

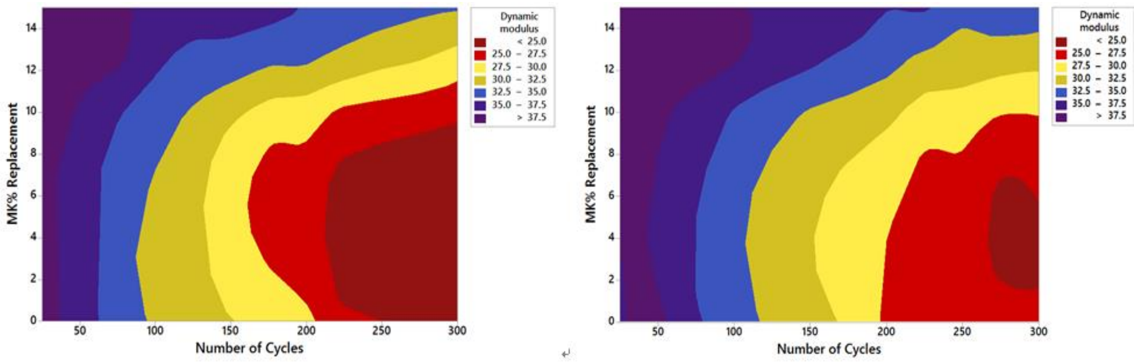
Mix Designs		Freeze and Thaw Cycles											
		25	50	75	100	125	150	175	200	225	250	275	300
Group 1	OPC	38.67	36.25	33.73	32.26	31.45	30.11	29.24	28.04	25.45	24.99	24.55	24.12
	MK10	38.41	37.24	35.26	33.63	32.26	30.62	29.15	28.76	27.11	26.60	26.13	25.56
	MK12.5	39.73	38.49	37.81	35.50	34.31	33.87	32.90	32.77	31.64	30.61	30.04	28.95
	MK15	39.65	39.53	38.01	36.65	36.17	36.38	35.95	34.82	34.08	33.71	33.01	32.74
Group 2	OPC	37.14	38.02	35.27	33.75	31.98	30.78	29.68	27.14	25.55	26.19	25.66	25.36
	MK10	39.49	38.09	36.44	34.87	33.49	32.33	31.60	30.63	29.05	28.73	27.69	27.69
	MK12.5	41.01	40.36	38.75	37.65	36.40	35.38	34.43	33.38	31.91	31.43	31.03	30.70
	MK15	40.46	38.91	37.52	38.00	36.52	35.93	36.39	35.09	35.30	32.99	34.32	34.08

MK up to 15% would not increase compressive strength substantially compared to control sample in Group 1. While by replacing 15% MK the compressive strength is increased in samples meaningfully and there is a significant difference between the samples containing MK and the control sample. The addition of MK in Group 2 caused a significant difference in compressive strength rather than Group 1.

3.3 Transport tests

For measuring transport performance, the water absorption and capillary absorption of the concrete samples containing MK were measured at different time intervals. The results confirmed that the percentage of water absorption and the height of capillary absorption are reduced by applying the MK. The performance of group 2 was slightly better than group 1 in absorption. The reason is attributed to improvement in the Interfacial Transition Zone (ITZ) of group 1 due to a reduction in total specific pore volumes of concretes. As it is shown in Table 6, increasing the curing time and percentages of MK may lead to a reduction in permeable gel pores due to the great bridging and filler effects.

3.4 Freeze and thaw



(a) Group 1 (b) Group 2

Fig. 5 Dynamic modulus of elasticity contour

Visual observations indicated that honeycomb voids were found on specimens as freeze and thaw cycles continued and these voids caused the mass loss. The weight was recorded for all the specimens during the freeze and thaw operations. One factor to evaluate the damage in concrete specimens subjected to the freeze and thaw cycles was a mass loss ratio. The relationship between the mass loss ratio of different specimens is plotted in Fig. 4.

As shown in Fig. 4, the amount of mass loss was higher in group 1. It indicates that higher water-to-binder-ratio likely increased the freeze and thaw degradation. Lower water-to-binder-ratio (under the same and adequate curing condition) decreased the volume of capillary pores and the permeability of the concrete. It prevented more damages, and thus, resulted in better freeze-thaw resistance. Moreover, it is illustrated that the mass loss increased gradually for all the concrete specimens as the freeze-thaw cycles continue. The highest mass loss was 4.51% for a control sample, and the lowest mass loss was 0.62% for 15% MK replacement with 0.4 water-to-binder-ratio. This might be attributed to a denser microstructure in the MK, due to the smaller size of MK particles, which might lead to less damage and less mass loss within the concrete.

Dynamic modulus of the elasticity of the different mixture samples was measured at constant intervals for up to 300 freeze and thaw cycles. As seen in Table 7, the mixtures with MK replacements displayed a slight decrease in the dynamic modulus of elasticity throughout the freeze and thaw test as opposed to what happened with the control specimen. Compared to the initial condition of samples before freeze and thaw cycles, the control sample showed a decrease 37.6% in the dynamic modulus of elasticity at 300th cycles, while the specimen with the 10, 12.5, and 15% MK showed 33.4, 27.1, and 17.4% decrease in the dynamic modulus of elasticity, respectively. These reductions were smaller in Group 2, which had less water-to-cement-ratio. The dynamic modulus of elasticity data is presented in Table 7.

By plotting the dynamic modulus contour, the effectivity of MK replacements is explained in Fig. 5 versus number of cycles. It was found that using more MK kept the dynamic modulus in a higher value. In the greater number of cycles, the higher MK replacement showed the higher dynamic modulus values. By comparing two w/b, less water to cementitious ratio led to a higher dynamic modulus in the same cycle.

The relative dynamic modulus of elasticity is the ratio of the dynamic modulus at a particular interval, relative to the dynamic modulus at the start of the test. Fig. 6 displays the relative dynamic modulus of elasticity data during the freeze and thaw cycles. The obtained data for

Table 8 Dynamic modulus of elasticity correlation coefficients

No.	Mixture Design	k	t	R2	The decrease in dynamic modulus of elasticity (%)
Group 1	OPC	2.104	-0.203	0.916	37.62
	MK10	1.940	-0.184	0.921	33.45
	MK12.5	1.563	-0.125	0.915	27.14
	MK15	1.347	-0.080	0.864	17.42
Group 2	OPC	2.019	-0.194	0.915	31.71
	MK10	1.752	-0.155	0.926	29.89
	MK12.5	1.614	-0.130	0.915	25.14
	MK15	1.307	-0.071	0.849	15.78

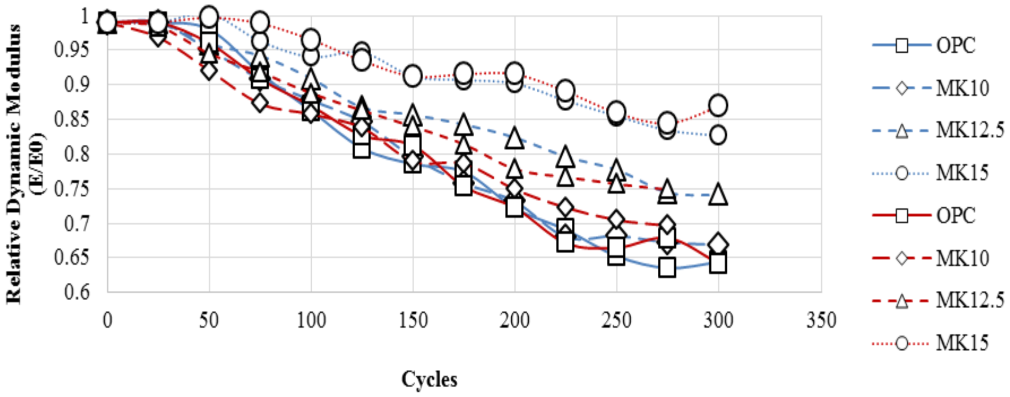


Fig. 6 Evolution of dynamic modulus of elasticity of concrete subjected to freeze and thaw cycles

specimens have been fitted to the function of the number of freeze and thaw cycles. The finest fits for samples have been acquired with a power equation (Eq. (4)).

$$E_n/E_0 = k \times n^t \tag{4}$$

where n is the number of freeze-thaw cycles, k and t are the coefficients. Table 8 summarizes all the test data on the dynamic modulus of elasticity and correlation coefficients.

3.5 Surface resistivity (SR)

The concrete surface resistivity was used as an indicator for concrete penetration. As shown in Fig. 7, specimens with MK had a higher electrical resistivity compared to the control concrete, with the rate about 3 times higher for the 15% replacement. The electrical resistivity was increased in group 2 with having lower the w/cm ratio. The maximum value of electrical resistivity was 62.5 kΩ.cm for the MK15 mixture with w/cm of 0.37 after 180 days. Also, the minimum was 14.9 kΩ.cm for the control specimen with 0.47 w/cm. The improvement of the concrete electrical resistivity was reported in previous studies (Tafraoui *et al.* 2016).

3.6 Water penetration test

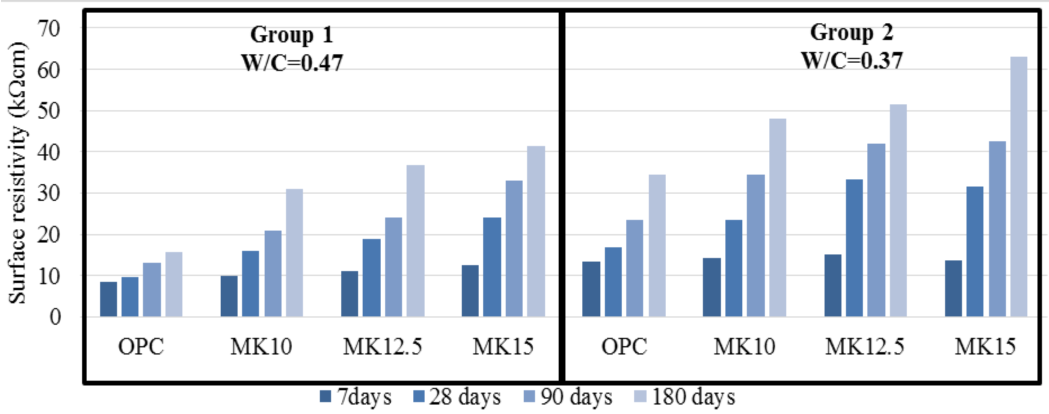


Fig. 7 The effect of metakaolin on the Surface resistivity (kΩcm) at various ages

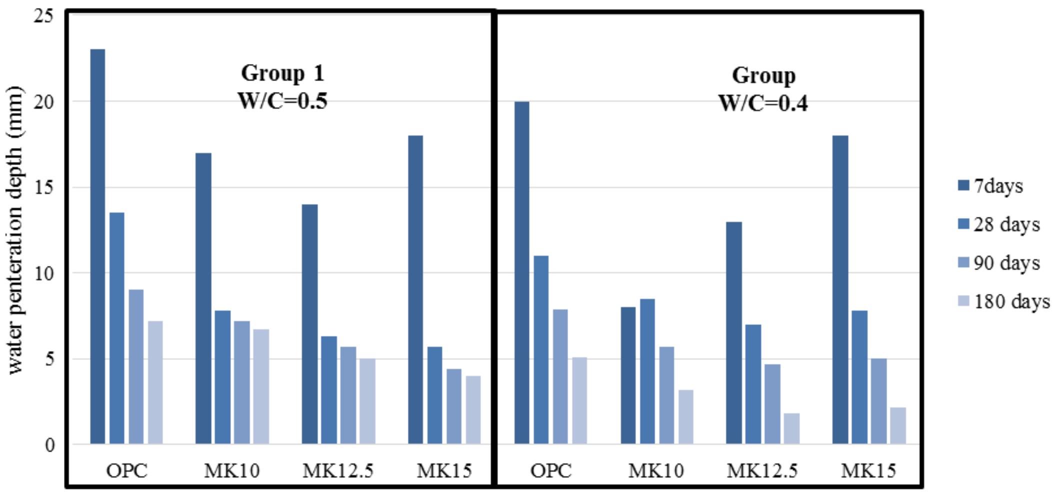


Fig. 8 The effect of metakaolin on the water penetration depth (mm) at various ages

Permeability is known to be one of the most important aspects of concrete durability. Lower permeability decreases the probability of chemical attacks due preventing soluble salts, such as chloride ions. Fig. 7 displays the results of the water penetration depths. The depth of penetration decreased for all mixtures with time and the MK incorporation resulted in more water penetration depth reduction due to to the filler effect, the pozzolanic reaction, and the heterogeneous nucleation (ASTM C33/C33M-16e1). In group 2, MK with 12.5% replacement had the lowest water penetration depth, 1.9 mm. In higher w/cm, group 1, the lowest penetration depth belonged to MK15. The cause of this change might be the clinker dilution effect.

3.7 Rapid chloride permeability test (RCPT)

The results for chloride permeability are shown in Fig. 8. This measurement was carried out with passed electric charge through the concrete specimens at the ages of 7, 28, 90 and 180 days. Based on Fig. 8, passing time and decreasing w/cm would reduce the charge. Results indicated that

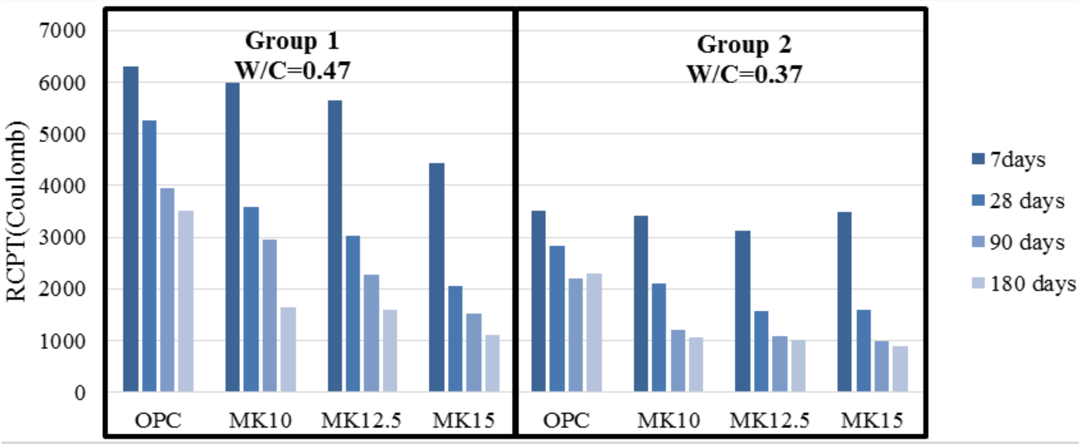


Fig. 9 The effect of metakaolin on the rapid chloride ion permeability (Coulomb) at various ages

specimens having MK meaningfully improved the resistance to chloride penetration. Same trends were observed in the previous study about the contribution of MK in chloride permeability (Ferreira *et al.* 2016). This development in resistance to chloride penetration is attributed to the pozzolanic reaction of MK with $Ca(OH)_2$. As shown in Fig. 8, at the age of 180 days, the maximum charged value in group 1 was 3506 coulombs, which was for control specimen. However, the same number for group 2 was 2291 coulombs. Based on ASTM C 1202, charge which was below 1000 coulombs, is considered as very low chloride permeability. Only specimens with 12.5% and15% MK were met this requirement and classified as "very low".

4. Conclusions

In this study, the effect of metakaolin (MK) as supplementary cementing materials and filling materials on the strength and durability of concretes was investigated. From the results obtained in this study, the following conclusion can be drawn:

• According to slump flow test results, the higher the replacement of cement by MK, the more SP was required to achieve the target slump flow, which probably stemmed from a significant amount of pores in the frame structure. The high surface area of MK and in turn leads to increased water demand.

• Concrete incorporating a new MK had higher compressive strength at various ages up to 180 days when compared with the ordinary Portland cement (OPC) concrete. The level of compressive strength developed with the period of curing and with decreasing the w/b ratio. For the materials in this study at w/b ratio 0.4, the optimum replacement of MK was 12.5% that is in agreement with former studies (Qian *et al.* 2001, El-Din *et al.* 2017).

• MK usage resulted in more water penetration depth reduction due to the filler effect, the pozzolanic reaction and the heterogeneous nucleation. Same as compressive strength test results, 12.5% was found to be the optimal level of incorporation in case of water penetration.

• Lower water-to-binder-ratio decreased the volume of capillary pores and the permeability of the concrete. It prevented more damages, and thus, resulted in better freeze-thaw resistance. Also, the second group of mixtures experienced less mass loss and dynamic elasticity modulus loss. In a study conducted by Kim *et al.* 10% replacement of MK was found to be optimal in the case of freeze-thaw resistance of mixtures with W/C=0.25 (Kim *et al.* 2007). However, in this study samples prepared by W/C=revealed the highest dynamic elasticity after freeze-thaw cycles, when 12.5% cement was replaced by MK.

• According to rapid chloride permeability test (RCPT), use of MK significantly enhanced the resistance to chloride penetration when compared with the OPC concrete. This improvement increased with increasing MK content. Same results were obtained by former studies (Poon *et al.* 2006, Bu *et al.* 2016)

• Results of the Surface Resistivity (SR) tests show that using MK drastically enhanced the electrical resistivity in comparison with the OPC concrete at about 2-4 times higher for the 15% MK mixture.

References

Ardalan, R.B., Joshaghani, A. and Hooton, R.D. (2017), "Workability retention and compressive strength of self-compacting concrete incorporating pumice powder and silica fume", *Constr. Build. Mater.*, **134**, 116-122.

ASTM C138/C138M-16a (2016), *Standard Test Method for Density (Unit Weight)*, Yield, and Air Content (Gravimetric) of Concrete, ASTM International, West Conshohocken, Pennsylvania, U.S.A.

ASTM C143/C143M-15a (2015), *Standard Test Method for Slump of Hydraulic-Cement Concrete*, ASTM International, West Conshohocken, Pennsylvania, U.S.A.

ASTM C150/C150M-16e1 (2016), *Standard Specification for Portland Cement*, ASTM International, West Conshohocken, Pennsylvania, U.S.A.

ASTM C33/C33M-16e1 (2016), *Standard Specification for Concrete Aggregates*, ASTM International, West Conshohocken, Pennsylvania, U.S.A.

ASTM C666/C666M-15 (2015), *Standard Test Method for Resistance of Concrete to Rapid Freezing and Thawing*, ASTM International, West Conshohocken, Pennsylvania, U.S.A.

Badogiannis, E.G., Sfikas, I.P., Voukia, D.V., Trezos, K.G. and Tsivilis, S.G. (2015), "Durability of metakaolin self-compacting concrete", *Constr. Build. Mater.*, **82**, 133-141.

Brykov, A., Krasnobaeva, S. and Mokeev, M. (2015), "Hydration of portland cement in the presence of highly reactive metakaolin", *Mater. Sci. Appl.*, **6**(5), 391.

BS 1881-116 (1983), *Testing Concrete. Method for Determination of Compressive Strength of Concrete Cubes*.

Bu, J. and Tian, Z. (2016), "Relationship between pore structure and compressive strength of concrete: Experiments and statistical modeling", *Sādhanā*, **41**(3), 337-344.

El-Din, H.K.S., Eisa, A.S., Aziz, B.H.A. and Ibrahim, A. (2017), "Mechanical performance of high strength concrete made from high volume of metakaolin and hybrid fibers", *Constr. Build. Mater.*, **140**, 203-209.

Ferreira, R.M., Castro-Gomes, J.P., Costa, P. and Malheiro, R. (2016), "Effect of metakaolin on the chloride ingress properties of concrete", *KSCE J. Civil Eng.*, **20**(4), 1375-1384.

Geng, H.N. and Li, Q. (2017), "Water absorption and hydration products of metakaolin modified mortar", *Key Eng. Mater.*, **726**, 505-509.

Ilić, B., Radonjanin, V., Malešev, M., Zdujić, M. and Mitrović, A. (2017), "Study on the addition effect of metakaolin and mechanically activated kaolin on cement strength and microstructure under different curing conditions", *Constr. Build. Mater.*, **133**, 243-252.

Johari, M.M., Brooks, J.J., Kabir, S. and Rivard, P. (2011), "Influence of supplementary cementitious materials on engineering properties of high strength concrete", *Constr. Build. Mater.*, **25**(5), 2639-2648.

Joshaghani, A., Ramezanianpour A.A. and Rostami, H. (2016), "Effect of incorporating sugarcane bagasse ash (SCBA) in mortar to examine durability of sulfate attack", *Proceedings of the 2nd International*

Conference on Concrete Sustainability, Madrid, Spain.
Kim, H.S., Lee, S.H. and Moon, H.Y. (2007), "Strength properties and durability aspects of high strength concrete using Korean metakaolin", *Constr. Build. Mater.*, **21**(6), 1229-1237.
Kurt, M., Kotan, T., Gül, M.S., Gül, R. and Aydin, A.C. (2016), "The effect of blast furnace slag on the self-compactability of pumice aggregate lightweight concrete", *Sadhana*, **41**(2), 253-264.
Piasta, W. and Zarzycki, B. (2017), "The effect of cement paste volume and w/c ratio on shrinkage strain, water absorption and compressive strength of high performance concrete", *Constr. Build. Mater.*, **140**, 95-402.
Poon, C.S., Kou, S.C. and Lam, L. (2006), "Compressive strength, chloride diffusivity and pore structure of high performance metakaolin and silica fume concrete", *Constr. Build. Mater.*, **20**(10), 858-865.
Qian, X. and Li, Z. (2001), "The relationships between stress and strain for high-performance concrete with metakaolin", *Cement Concrete Res.*, **31**(11), 1607-1611.
Ramezanianpour, A.A., Mahdikhani, M. and Ahmadibeni, G. (2009), *The Effect of Rice Husk Ash on Mechanical Properties and Durability of Sustainable Concretes*.
Rezaifar, O., Hasanzadeh, M. and Gholhaki, M. (2016), "Concrete made with hybrid blends of crumb rubber and metakaolin: Optimization using response surface method", *Constr. Build. Mater.*, **123**, 59-68.
Salau, M.A. and Oseafiana, O.J. (2015), "Effects of temperature on the pozzolanic characteristics of metakaolin-concrete", *Phys. Sci. Int. J.*
Siddique, R. and Khan, M.I. (2011), *Supplementary Cementing Materials*, Springer Science & Business Media.
Tabatabaeian, M., Khaloo, A., Joshaghani, A. and Hajibandeh, E. (2017), "Experimental investigation on effects of hybrid fibers on rheological, mechanical, and durability properties of high-strength SCC", *Constr. Build. Mater.*, **147**, 497-509.
Tafraoui, A., Escadeillas, G. and Vidal, T. (2016), "Durability of the ultra high performances concrete containing metakaolin", *Constr. Build. Mater.*, **112**, 980-987.
Türkel, S. and Altuntaş, Y. (2009), "The effect of limestone powder, fly ash and silica fume on the properties of self-compacting repair mortars", *Sadhana*, **34**(2), 331-343.
Yousuf, F., Wei, X. and Tao, J. (2017), "Evaluation of the influence of a superplasticizer on the hydration of varying composition cements by the electrical resistivity measurement method", *Constr. Build. Mater.*, **144**, 25-34.

JK